Position Resolved Diffraction of Tooth and Bone: Sub-millimeter to Sub-micrometer

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Bones and tooth contain a composite material: a collagen matrix reinforced by a high density of hydroxyapatite (hAp) nanoplatelets. These mineralized tissues obtain their remarkable mechanical performance through the interplay of a hierarchy of structures. X-ray scattering has contributed significantly to the understanding of changes in bulk characteristics of bone (e.g., changes in hAp lattice parameters, microstrain and crystallite size accompanying aging), but position resolved studies are required to come to grips with interactions between the various micro- and nano-structural scales. The examples in this presentation illustrate the how different x-ray probe characteristics are needed depending on the scales being studied.

Dentin forms the bulk of tooth volume in most mammals, and a characteristic feature of dentin is its array of aligned tubules. Thin collars of peritubular dentin (PTD, < 1 μm thick) surround tubule lumens in many species, and intertubular dentin (ITD) provides the bulk of the tissue. The structural and compositional differences between PTD and ITD remain poorly understood, and simultaneous diffraction and fluorescence mapping with a 250 nm wide beam have revealed differences in hAp crystallographic texture and in Zn and Ca composition.

Cementum is the tooth tissue to which the periodontal ligaments attach, new cementum layers are added every year and this tissue normally does not remodel. This “recording structure” is visible optically and used to determine an animal’s age and even the season at death. The changing biomineralization processes remain obscure, and fluorescence plus diffraction mapping have been used to understand seasonal changes in biomineralization of cementum from whale teeth, where the layers are > 100 μm wide, and from reindeer and bovine teeth where layer width is ~ 10 μm.

The preceding examples utilized sectioned materials, and often sectioning is not allowed or diffraction maps in 3D are required. Scattered x-radiation can be used for computed tomographic reconstruction of the distribution of crystallographic phases within the interior of specimens, and diffraction patterns can be measured for each volume element (voxel) within a reconstructed slice. Results are presented for specimens containing: a) different materials (SiC/Al composite); b) different polytypes (calcite/aragonite in a bivalve attachment system); c) mixtures of nanocrystalline and amorphous phases; d) a single phase, but volumes with different hAp lattice parameters; e) a single phase containing a spatial distribution of crystallographic texture (bone) and f) a single phase with a spatial distribution of strains produced by in situ loading (bone).

Position-resolved x-ray scattering has also been applied to intact human second metacarpal (Mc2) bones from Roman era and from medieval era Britain. Destructive sampling is not an option, and the > 10 mm bone diameter, irregular cross-section and large hAp *d*-spacings combine to make this a challenging diffraction problem. Results from two Mc2 from each of three age (at death) cohorts are compared with those from a modern Mc2 and two synthetic hydroxyapatite (hAp) phantoms designed to reveal geometrical effects. We investigate how much diagenesis has altered the bone (changed hAp lattice parameters; crystallite size, microstrain; collagen D-period). We see small diagenetic changes and study whether the above quantities change with age in each of the two populations and whether these changes are similar to those observed for bone from modern sedentary populations.